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Adaption and Implementation of a MgO-Lime-$CaCl_2$ based Defluoridation System for Use in Rural Rajasthan

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1. Introduction

Fluorine is 13th most abundant element available in earth crust. It is a member of the halogen group having atomic number 9 and molecular weight 19. It has most electronegative of all elements and exist only diatomic molecule,

Fluorine is a highly reactive gaseous element belonging to the VIIth group of the 2nd period of the periodic table. In the earth crust its natural abundance is 0.6 to 0.9 ppm. Although chlorine is the most electronegative element, the electron affinity of fluorine is lower than that of chlorine, this is because of the smaller size of the fluorine atom and hence higher electron density which makes the addition of an extra electron relatively difficult in case of fluorine than chlorine[1].

Drinking water with excess fluoride causes fluorosis, which is a widespread disease in countries like India, Pakistan, Bangladesh, Argentina, United State of America, Kenya etc.[2] In India 62 million people are affected by fluorosis[3]. The most fluorosis affected states are Rajasthan, Andhra Pradesh, Karnataka, Tamil Nadu, etc.[4]In Rajasthan, all the districts are fluoride affected. The district of Nagaur, Jaipur, Sikar, Jodhpur, Barmer, Jhunjhunu, Churu, Bikaner are the worst affected by fluorosis.[5 6 7]

The origin of fluoride in the ground water of Rajasthan is the underground igneous rocks[8]. Large deposit of Flurospar [CaF_2], Fluoroapatite [$Ca_3(PO_4)Ca(FCl)_2$], Cryolite [Na_3AlF_6] and Mica [$(X_2Y_{4-6}Z_8\underline{O}_{20}(\underline{OH},\underline{F})_4$)(X- Na, K, Ca, Y-Al, Mg, Fe, X-Si,Al)] occur in this region[9]. Leaching of fluoride from this minerals is the main source of fluoride contaminated ground water. Depending upon the total amount of fluoride intake, fluorine may be beneficial or harmful to the human health.[2 10] For proper development of dental enamel, fluoride is beneficial, if present in the desirable range of 0.6 to 1.0 ppm.[11]Drinking water with fluoride above 1.5 ppm cause fluorosis, a crippling disease of bone and teeth.[12 13]

In aluminum industry, cryolite is used in the electrolytic extraction of aluminium from bauxite. Fluorine containing compounds are used in the production of polymers like Teflon (-CF_2-CF_2-), Polyvinlidene fluoride (-CF_2CH_2-), Polyvinylfluoride (-CH_2CHF-), and refrigerants (CFC).[14]Fluorine containing compounds like SF_6 is used as a gaseous insulator in voltage

transformers. Hydrofluoric acid is used for pickling (cleaning) stainless steel and etching glass. Various fluoride containing compounds like sodium fluoride and monofluroacetate[15] are potent insecticides, cyanofluoropyridine[16] is potent fungicide while sodium fluoroacetate[17] is a rodenticide.

2. Purpose of study

Drinking water contaminated with fluoride cause many serious health related problems like weakening of muscles and bones, premature ageing, impairment of nervous system, pain in joints, inability to do physical work and lowering of I.Q in children etc[18]. Many places of Laxmangargh in Sikar district of Rajasthan have high levels of fluoride and this has been conformed by recent studies[5]. Prevalence of fluorosis in common in this region and there is no defluoridation of drinking water in this place.

There are many methods using removing fluoride from water including the Nalgonda technique[8] [19], reverse osmosis[8], activated alumina,[8] [20] [21] [22] ion exchange,[8] [23] fly ash,[24] clay column.[2] [25] A new method of defluoridation using MgO-Lime-$CaCl_2$, and HCl has been developed at IISc Bangalore.[26] [27] This method has been successfully used in field trials in Kolar district in Karnataka.[28] [29] My research work would involve a suitable modification of this technique for removal of fluoride from local fluoride contaminated ground water taking into account the different water characteristics of Rajasthan and Bangalore above the method was originally developed. [30] [31] Field trials at the modified filter will be carried out in selected village of Laxmangargh in Sikar district of Rajasthan.[32]

3. Literature Survey

Fluoride has remarkably low dissociation energy (38 K cal/mole).[33] As a result it is highly reactive and has a strong affinity to combine with other elements to produce compounds known as fluorides. Drinking water with excessive concentration of fluoride causes fluorosis, which is a progressive disease and in the long run becomes crippling.[13] [14] Fluoride contamination of ground water occurs through a natural process in which fluoride bearing rocks are leached by ground water releasing fluoride.[34]

Fluoride is known to contaminate ground water reserves globally. As per a conservative estimate approximately 200 million people from more than 35 countries are at risk of fluorosis. The main fluoride affected countries are India, China, Tanzania, South Africa, Kenya, Ghana, parts of USA, Mexico, Ethiopia, etc. [2] In India fluoride contaminated ground water is prevailing in 19 states among them Rajasthan, Andhra Pradesh, Madhya Pradesh, Punjab, Gujarat, are the most fluoride affected.[35 36 37]

In India approximately 60 to 65 million people drink fluoride contaminated ground water out of which 2.5 to 2.6 million people are affected by fluorosis.[38 38] In India 177 district are affected by fluoride.[36] According to one estimate 6 million children are affected by fluorosis.[38 39]

Health affectes of fluoride, Skeletal fluorosis[1 22 39]

Prolonged intakes of water have fluoride □8mg/l leads to skeletal fluorosis, Bow legs and Knock Knees are the characteristic of the skeletal fluorosis. Restriction of spine movement is the earliest clinical sign, and the stiffness increase steadily until entire spine becomes a continuous column of bone. Changes in the bone structure can be detected by X-ray.

Concentration of fluoride in drinking water and its effect[5]

Concentration of Fluoride and fluoride in ppm	Health effect
0	Limited growth and fertility
□ 0.5	Dental caries
0.5 to 1.5	Good dental and Skeletal health
1.5 to 4.0	Dental fluorosis
4.0 to 10.0	Dental fluorosis and skeletal fluorosis
□ 10.0	Crippling fluorosis and acute fluoride toxicity

Non- Skeletal fluorosis[40]

Soft tissues and organs like muscles liver and kidney, blood and reproductive system are also affected by fluoride. Acute abdominal pain, constipation, diarrhea, nausea, loss of appetite, are common symptoms of fluoride affected people. Tingling sensation in toes and fingers, depression and nervousness are some signs of the Neurological manifestations of fluorosis. Its reported that fluoride lowers the I.Q of children's. It has been reported that in fluorosis affected areas people suffers from skin rashes, which appear and disappear by themselves. It has been absorbed that person affected by fluorosis have less volume of urine this may also due to the adverse affect of fluoride on the region of brain controlling urine production.Fluoride destroys the red blood cell by lowering the calcium content of its membrane, therefore low hemoglobin is usually reported in persons suffering fluorosis. Its known that fluoride disturbed the hormones system, irritate the respiratory tract, and lowers the metabolic activity of the people.

Fluoride Standards

Organization	Desirable Limits (ppm)	Permissible Limits (ppm)
WHO[41]	0.5 to 1.0	1.5
BIS[42]	1.0	1.5
ISO[43]	0.6 to 1.2	1.5
ICMR[44]	1.0	1.5
USPHS[45]	0.7	1.2

Fluoride Removal Techniques

Nalgonda Technique[8 20]

In the Nalgonda process calculated dosage of Alum and Lime is added to fluoride contaminated water with vigorous stirring. Aluminum hydroxide flocs are produced which settle down to the bottom and during the flocculation process, fluoride ions are partially removed by electrostatic attachment to the flocs.

HCO^- and silica are not present. Alumina (Al_2O_3) it's practically insoluble in water, its scarcely attacked by strong reagents. Alumina has to be activated for defluoridation process and different grade of activated alumina are available locally at low cost. The fluoride removal capacity of activated alumina depends upon the porosity and surface area of activated alumina. Both household and community level defluoridation can be carried out defluoridation activated alumina. In India this is most common defluoridation technology adopted and promoted by UNICEF. Activated alumina can be regenerated using NaOH and HCl. Activated alumina is amphoteric in nature and can exist as $AlOH_2^+$, AlOH and AlO^-. The following mechanism are usually used to describes fluoride removal.

$$AlOH_2^+ + F^- \rightarrow AlF + H_2O$$

$$AlOH + F^- \rightarrow AlF + OH^-$$

The activated alumina technology has following advantages: a) its effective and economical, b) it requires minimum contact time for maximum defluoridation capacity and it has high regeneration capacity.

This technology however has following disadvantages: a) regeneration of activated alumina is cumbersome process and can only be carried water to the help of trained people, b) residual alumina in water can be present, c) activated alumina may have to be regenerated in short time intervals and this method becomes ineffective, if the TDS of water exceeds 1500 ppm.

Clay column [2 26]

Clay bricks have been used in Sri Lanka for defluoridation purposes. Water is allowed to pass through vertical columns containing crushed clay bricks, which adsorbed fluoride. This method has following advantage: a) it's a low cost technique and the water after defluoridation, is clean and odourless, fit for human consumption. This method has following disadvantages: the defluoridation technique has not been carried out with different grade of clay available in different parts of the world, so its applicability may be limited.

The following reaction occur:

1. Aluminum dissolution

$$Al_2(SO_4)_3.18H_2O \rightarrow 2Al^{3+} + 3SO_4^{2-} + 18\ H_2O$$

2. Aluminum precipitation

$$2Al^{3+} + 6H_2O \rightarrow 2Al(OH)_3 + 6H^+$$

3. Co-precipitation

$$F^- + 2Al(OH)_3 \rightarrow \text{Al-F complex} + \text{undefined product}$$

4. pH adjustment

$$6Ca(OH)_2 + 12H+ \rightarrow 6Ca^{2+} + 18\ H_2O$$

The method has following advantages: a) it can be used at both domestic and community level filtration process, b) the chemicals are used are same as those urban and municipal water supply scheme, c) the method is cheap, d) the defluoridated water meets the standards of drinking water quality, e) no regeneration of the filter is needed required, f) the method is simple and requires low maintenance, g) unskilled labours can operate the filter, h) fluoride can be effectively removed from upto 20 ppm to less than 1.2 ppm and turbidity is also removed.

The method has followed disadvantages: a) the filter has operated by batch method and continuous manner, b) if the dosage of lime is exceeded then there is chances of contamination due to aluminum in treated water beyond the permissible limit of 0.2 ppm, c) the method requires high chemical dosage depending on the fluoride concentration, d) because of addition of aluminium sulfate the sulfate concentration may exceed above 400 ppm in water and safe disposal of sludge is problem in the Nalgonda technique.

Activated Alumina [8 21 22 23]

Activated Alumina is excellent adsorbent for the removal of fluoride from the drinking water of various compositions. The adsorption capacity of activated alumina is very favorable for treatment of water containing □ 20 ppm fluoride concentration, provided interfering ions like

Bone Charcoal Method [2 26]

Bone charcoal is prepared by heating bone at high temperature. Bone charcoal is mainly calcium hydroxylapatite [Ca_{10} $(PO_4)_6$ $(OH)_2$] where the hydroxyl groups are replaced by fluoride according to the following equation,

$$Ca_{10}(PO_4)_6(OH)_2 + 2F^- \rightarrow Ca_{10}(PO_4)_6F_2 + 2OH^-$$

This method has following advantages: a) if bones are available locally and then it is low cost method, b) the water after passing through bone charcoal has no odour, it is clean as fit for human consumption. This method has following disadvantages: a) if bone charcoal is not prepared properly, it may impart taste and colour to water, b) biological residue may be remain, which may leach out into water and the bone charcoal requires periodic regeneration and it is affected by alkalinity of the water, Further it may not be universally applicable and due to religious belief.

Reverse Osmosis [8]

In the Reverse osmosis process, water under pressure is passed through a semi permeable membrane, whereby most of the ions in water are retained including fluoride. Reverse osmosis method has following advantages : a) it can be remove bactria and viruses and, b) if there are other impurities in water like heavy metals they can also be removed. Reverse Osmosis method has following disadvantage: a) there is 60% wastage of water, b) the membrane has to be cleaned periodically and replaced at a high cost and this method is effected by turbidity of water.

Contact Precipitation [8 20]

In contact precipitation calcium and phosphate compounds are added to bone charcoal during defluoridation process. This method has following advantage: a) it is low cost method, If bone charcoal can be sourced locally and fluoride uptake capacity is increased due to present of calcium and phosphate compounds. This method has following disadvantage: the calcium phosphate cannot be regenerated and the exact chemicals process of defluoridation is not fully understood.

MgO-Lime-$CaCl_2$-$NaHSO_4$ Method [27 28 29 30]

The IISc method relies on magnesium oxide based, precipitation-sedimentation-filtration technique to reduce fluoride concentrations in water to permissible levels (< 1.5 ppm). It uses a domestic defluoridation unit (DDU) to implement the chemical treatment procedure. The magnesium oxide method for fluoride removal has several advantage. Magnesium oxide has very limited solubility in water and therefore does not add to total dissolved salinity. All chemicals used in the process are non-toxic. Alkaline pH of the magnesium oxide treated water is easily neutralized by addition of sodium bisulfate solution. The method does not involve any recharge process and thus avoids generation of corrosive and toxic wastes. The method is designed to treat fluoride-contaminated ground water with varied ionic compounds. The sludge can be converted into stabilized mud blocks for construction purposes..

Principle of the method

The method treats fluoride contaminated groundwater with fluoride ion concentration ranging from 1.5 to 7 mg/L. Fluoride ions are removed by magnesium oxide through chemisorptions mechanism. On addition to water samples, magnesium oxide hydrate to brucite [$Mg(OH)_2$] as,

$$MgO + H_2O \rightarrow Mg(OH)_2$$

During brucite formation, fluoride ions (F^-) in the contaminated water samples are incorporated into the brucite lattice by isomorphous substitution of hydroxyl ions by fluoride ions. The isomorphous substitution reaction leads to the formation of the $Mg(OH)_{2-y}.F_y$ phase. In addition to magnesium oxide, calcium chloride solution, sodium bisulfate and calcium hydroxide are added to the raw water. Addition of magnesium oxide increases the pH of water between 10.1 and 10.3. Sodium bisulfate solution reduces the pH of magnesium oxide treated water within potable water limits (6.5 to 8.5) by the reaction.

$$HSO_4^- + OH^- \rightarrow SO_4^{2-} + H_2O$$

Bicarbonate ions present in the fluoridebearing water samples get converted to carbonate ions in the alkaline pH created by magnesium oxide addition according to the reaction.

$$HCO_3^- + OH^- \rightarrow CO_3^{2-} + H_2O$$

The carbonate ions formed formed in the above reaction, in turn consume an additional equivalent of sodium bisulfate as,

$$CO_3^{-} + HSO_4^{-} \rightarrow HCO_3^{-} + SO_4^{2-}$$

Calcium hydroxide and calcium chloride solutions reduce the interference of bicarbonate ions towards sodium bisulfate consumption by transformation of soluble bicarbonate to insoluble calcium carbonate as follows:

$$HCO_3^{-} + Ca^{2+} + OH^{-} \rightarrow CaCO_3 + H_2O$$

Chemical treatment procedure

Known weights of Magnesium oxide-Calcium Hydroxide mix + known volume of 7.5 % Calcium Chloride solution (table 1) are added to fixed volumes (50 or 100 liters) of fluoride contaminated water. Efficient contact between added chemicals and fluoride (in water) is achieved by stirring the fluoride contaminated water for 3-5 minutes using electrical or hand operated stirrer. The water is allowed to settle for 16 hours at the end of which the Magnesium Oxide sludge settles to the bottom of container. The clear water is filtered into another container for pH adjustment using $NaHSO_4$ and the water is fit now for human consumption. The Magnesium Oxide sludge is carefully stored in a 100 liter container till further use.

Table.1

Initial fluoride concentration, mg/L	Volume of 7.5 % calcium chloride solution to be added, ml/L	Magnesium oxide dosage, g/L	Initial bicarbonate concentration, mg/L	Calcium hydroxide dosage, g/L	Volume of 5 % sodium bisulfate solution to be added to treated water, ml/L
1.5-7	4	1-1.5	250-400	0.3	1-3
1.5-7	4	1-1.5	>400-500	0.4	1-3
1.5-7	4	1-1.5	>500-700	0.5	3-6

Why we have chosen the Magnesium Oxide- Lime- Sodium Bisulfate defluroidation method?

a) All chemicals used are non-toxic, b) Chemicals are easily available in powder form, c) Easy dosage of chemicals, d) No chemicals in taste of treated water, e) No residual aluminium in water, which is reported to cause Alzheimer's disease, f) No disposal problem of waste washings, g) No recharge of filter material is involved, h) Less amount of sludge is produced, i. The sludge can be disposed off safely by converting it into building materials, j) The method can even be carried out without any special filter mechanism, k) The technique can be scaled up easily to treated any amount of water.

4. Proposed Work

In the proposed research work the defluoridation method developed at IISc Bangalore using lime, MgO, $CaCl_2$, and $NaHSO_4$, will be suitably modified in the laboratory, to suit the ground water characteristic of Laxmangarh, in the district Sikar of Rajasthan[31 32]. The ground water in this region has high level of alkalinity in the order of 1000-1500 ppm. Our initial studies have shown that pH adjustment with $NaHSO_4$ is not suitable has the pH adjusted water has TDS exceeding the permissible limit of 2000 ppm, also the large volumes of 5% $NaHSO_4$ solution has to be added in order to be neutralized. The high concentration of carbonate ions in the water after 16 hrs this increases the sulphate concentration above the permissible limit of 400 ppm, therefore pH adjustment with HCl would be used. The permissible limit of chloride in this case is much higher at 1000 ppm. The dose adjustment of MgO, Lime, and $CaCl_2$ would be a challenge as the local has this water has low calcium concentration and high bicarbonate concentration. Also the dose adjustment of HCl has to be done keeping in mind the corrosive nature of concentrated HCl and large volume requirement of dilute HCl, which would cause handling problems in village during filed trials. Initial studies have shown the nitrate concentration (approximately 100 ppm) is above the permissible limits. This method dose not reduce nitrate concentration, also since nitrate is been added from outside, the nitrate concentration will remain unaffected. Field trials of the filter will be conducted village Shola of Laxmangarh Teshil[33]. The willing villages will be trained to operate filters themselves. The performance of filter will be monitored and investigation will be done in the conversion of waste sludge into building materials.

5. METHODOLOGY

1. Collection of water samples and analysis

Initial field survey will be done to collect water samples and measure the concentration of F^-, pH, TDS, CO_3^{2-}, HCO_3^-, Cl^-, Na^+, K^+, SO_3^{2-}, NO_3^-.

2. Optimisation of chemical dosage for the defluoridation filter

(a) Optimisation of MgO dosage

Several 1 liter fluoride contaminated ground water samples would be treated with various quantities of MgO and fixed dosage of CaO and fixed volume of 7.5% $CaCl_2$ (in demineralised water). The mixture would be stirred for five minutes and left overnight for 16 hrs. The white floc which settles at bottom will be filtered on the next day. The portion of filtrate will be set aside for analyses and a fixed volume of the filtrate will be treated with dilute HCl, till the final pH of the water would be between the permissible limit of 6.5 to 8.5. Different volumes of HCl would be required in each case. The pH adjusted HCl treated water would be analysed for various water quality parameters like F^-, pH, TDS, CO_3^{2-}, HCO_3^-, Cl^-, etc. The optimum dose of MgO which shows minimum residual fluoride concentration in pH adjusted water would be identified. The other water quality parameters of the pH adjusted HCl treated water, at this optimum dose of MgO, would be reviewed with respect to their permissible limits in drinking water.

(b) Optimisation of $CaCl_2$ dosage

Several 1 liter fluoride contaminated ground water samples would be treated with varying volumes of 7.5% $CaCl_2$ (in Deminaralised water), and optimized dosage of MgO and fixed dosage of CaO would be added to each of the samples. The mixture would be stirred for five minutes and left overnight for 16 hrs. The white floc which settles at bottom will be filtered on the next day. The portion of filtrate will be set aside for analyses and a fixed volume of the filtrate will be treated with dilute HCl, till the final pH of the water would be between permissible limit of 6.5 to 8.5. Different volumes of HCl would be required in each case. The pH adjusted HCl treated water would be analysed for various water quality parameters like F^-, pH,

TDS, CO_3^{2-}, HCO_3^-, Cl^-, etc. The optimum dose of $CaCl_2$ which shows minimum residual fluoride concentration in pH adjusted water would be identified. The other water quality parameters of the pH adjusted HCl treated water, at this optimum dose of $CaCl_2$ would be reviewed with respect to their permissible limits in drinking water.

(C) Optimisation of CaO dosage

Several 1 liter fluoride contaminated ground water samples would be treated with optimized dosage of MgO and optimized volumes of 7.5% $CaCl_2$ (in Deminaralised water), and varying dosage of CaO would be added to each of the samples. The mixture would be stirred for five minutes and left overnight for 16 hrs. The white floc which settles at bottom will be filtered on the next day. The portion of filtrate will be set aside for analyses and a fixed volume of the filtrate will be treated with dilute HCl, till the final pH of the water would be between permissible limit of 6.5 to 8.5. Different volume of HCl would be required in each case. The pH adjusted HCl treated water would be analysed for various water quality parameters like F^-, pH, TDS, CO_3^{2-}, HCO_3^-, Cl^-, etc. The optimum dose of CaO which shows minimum residual fluoride concentration in pH adjusted water would be identified. The other water quality parameters of the pH adjusted HCl treated water, at this optimum dose of CaO would be reviewed with respect to their permissible limits in drinking water.

3. Fabrication of Filter

The defluoridation filter would consist of 2 units of 50 liter each made of food grade (HDPE). A tap would be fixed 2 cm above the bottom of 50 liter container which would serve as precipitation, coagulation, sedimentation, filtration unit. In the second container pH adjustment with HCl will take place, and this would also serve as storage tank for defluoridated water. For stirring of the chemicals with water in first container a food grade U-PVC pipe would be used. For filtration purpose, six layers of white cotton cloth would be used.

5. Field trials of the filter

Filter will be installed that willing house hold in the village Shola etc. The dosage of chemical used would be the optimized dosage from the laboratory. The villagers would be trained on use of filter, any operational problems of the filters will be resolved during the field trials. The performance of the filter would be closely monitored and compared with lab trials conducted earlier.

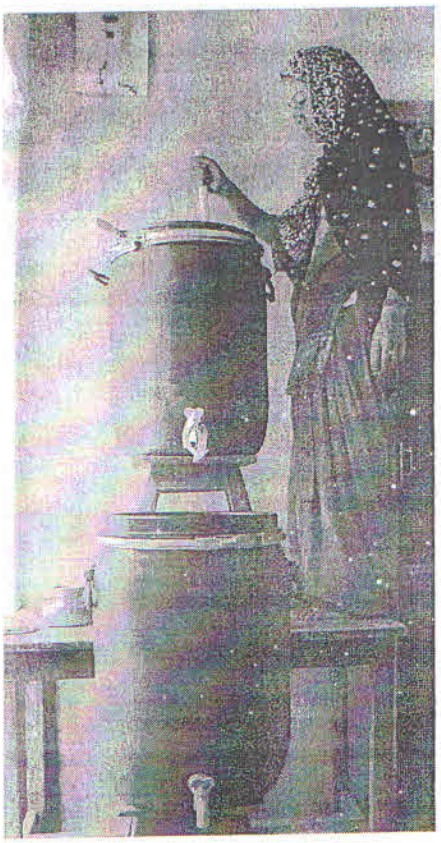

Figure.2 Photograph of filter during field trials in village Pillani Ki Dhoni in Shola, Laxmangarh.

Figure.1 Photograph of filter during lab trials

4. Laboratory testing of fabricated filters

Large quantity of water samples will be transported using mini tankers of 500 liter capacity and stored in lab. The laboratory trials of the filters would be conducted using the fluoride contaminated ground water brought from village and optimized dosage of MgO, $CaCl_2$, CaO and HCl. For a particular tube well the process will be repeated using several batches of water. The water quality parameters would be determined and adjustment of dose of chemicals would be made if necessary.

Figure.3 Photograph of filter during field trials in village Kumar Ki Dhoni in Shola, Laxmangarh.

6. Conversion of sludge into stabilized mud blocks

1 Determination of optimum water content of clay bricks.

Clay (sourced locally) would be mixed with varying volumes of water and will be made into stabilized mud blocks using stabilized earth block making machine. After curing the compressive strength of the mud block would be determined. The water content of the brick with highest compressive strength will be the optimum water content.

2 Determination of optimum water content of clay and 8% cement bricks.

Bricks of the above mentioned composition would be made with varying water content and after curing the compressive strength would be determined. The water content of the bricks with highest compressive strength will be the optimum water content.

3 Determination of optimum water content of clay and 8% cement and 10% sludge

Bricks of the above composition with varying water content will be made and compressive strength measured after curing.

4 Fluoride leaching test for bricks containing sludge.

The bricks with highest compressive strength would be chosen for fluoride leaching test. The powered bricks would be subjected to fluoride leaching using distilled water and TCLP extractant. The fluoride leaching from bricks will be reviewed with respect to the permissible limit of fluoride in drinking water.

5 Twenty five filters will be installed in the village after due chemical dosage optimization and lab trials.

6 All the above the data from various studies will be compiled in the form of a Ph.D thesis.

6. Work already done

Out of the 25 filter to be installed 10 filters have been already installed. In this 10 filters six are currently in operation, other filters will be installed soon, after dosage optimization.

7. Conclusion

Initial studies have shown that the MgO-Lime-$CaCl_2$-$NaHSO_4$ method is not suitable in Rajasthan due to the higher alkalinity of the ground water here. The method has been modified by us and HCl is used instead of $NaHSO_4$. If this is not done, the SO_4^{2-} in the defluoridated water is more than the permissible limit of 400 ppm. For each tube well with varying fluoride concentration, the dosage optimisation studies are being carried out in lab, after which the filter are being put to field trials.

8. Results obtained

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